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**Naval Surface Warfare Center**
Indian Head, MD 20640-5035

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# RECOVERY TECHNIQUE FOR SHOCKED EXPLOSIVE SAMPLES

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**13. ABSTRACT** (*Maximum 200 words*)

A technique has been developed to recover explosives that have undergone stresses as high as 26 kilobars. Physical (microscopic) and chemical examination of recovered explosives has led to the discovery of new chemical reactions occurring in these explosives.

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# FOREWORD

This work was performed for and funded by the Office of Naval Research as part of the Explosives Project within the Explosives and Undersea Warheads Technology Block Program PE602314N. The results and conclusions in this report will be of interest to those seeking information on (1) shock wave sensitivity of explosives, (2) chemistry of recovered shocked explosive samples, and (3) surface chemistry techniques.

The authors wish to acknowledge Jack Marshall for the design of the aluminum frames which held the donor pentolite sphere and samples in place prior to detonation of the donor. Dr. Harold Sandusky and Carl Groves prepared the recovery capsules containing the RDX and CL-20 crystals for experiment 92-R1. Cynthia Forbes typed this report.

Approved and released by:

Ruth Doherty
Head, Detonation Physics Division

## CONTENTS

## INTRODUCTION

Pre-ignition reactions in explosives subjected to shock compression are of fundamental importance to the study of explosive behavior. Recovery of samples subjected to strong shocks has been a problem. The sample must be contained and remain relatively uncontaminated. Typically, attempts at recovery have resulted in the physical destruction of the explosive test samples due to strong rarefactions and violent collisions with objects such as chamber walls. In addition to the recovery of the explosive sample, some means of determining the shock history in the sample should be available. In most recovery techniques, the impedances of the surrounding materials are quite different from the explosive samples and the confinement is finite in size. Both of these conditions lead to relief waves that significantly affect the strain histories of recovered samples. The use of materials with similar impedances greatly reduces the magnitude of reflected waves within the sample.

In our technique, small (usually 30 mg) explosive samples, encapsulated in Teflon holders, are subjected to strong shock compression. The shock-producing system is the same as that used in the Underwater Sensitivity Test (UST).[1,2] The underwater shock system was carefully calibrated previously.[3] The recovered explosive samples are then removed from the capsules for chemical and physical (microscopic) analysis. Recovery of samples shocked to peak stresses of up to 26 kbar with pulse widths of a few microseconds has been accomplished. The present recovery technique is the result of a number of experiments in which various degrees of success occurred. Modifications after each experiment finally led to a reliable recovery technique.

## EXPERIMENTAL TECHNIQUE FOR THE RECOVERY SYSTEM

**Donor and Detonator:**

In the recovery system, the donor is an 82-mm-diameter sphere of cast pentolite (50% TNT/50% PETN) weighing 470 to 480 g. The spherical charge assembly is shown in Figure 1. The detonator, an RP-80, is an exploding bridgewire type manufactured by Reynolds Industries Systems Inc. It is 7.11 mm in diameter and fits into a 46-mm-deep hole cast (not machined) in the sphere. The RP-80 detonator is insensitive to static discharge and requires at least a 1.0-μF capacitor charged to 2.5 kV to initiate detonation in the detonator. This makes it quite safe for inserting into the pentolite sphere. A 7.0-mm-diameter by 9.5-mm-long pellet of pressed pentolite (density = 1.6 g/cm$^3$) is inserted in the hole ahead of the detonator to ensure a detonation at the center of the cast pentolite sphere. (Pressed pentolite is much more sensitive to shock than is cast pentolite, the run distance to detonation being negligible and the propagation of detonation being essentially isotropic.) The available space around the detonator leads within the hole is filled with C-4 plastic explosive. A sealant (Duxseal) is used to cover the connection of the plastic sheath, containing the detonator leads, to the pentolite sphere.

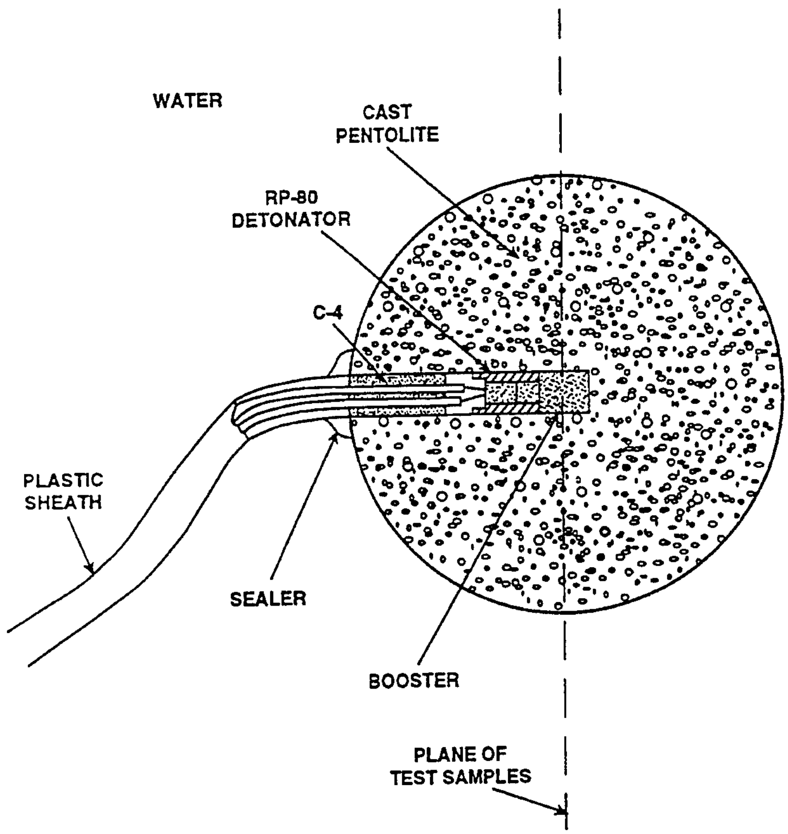



FIGURE 1. SPHERICAL DONOR CHARGE ASSEMBLY

**Placement of Components in Tank:**

The spherical donor is suspended by a nylon cord harness in a cubic tank of water, 60 cm on an edge. The harness is attached to an aluminum supporting frame. The test samples enclosed in Teflon are mounted in thin-walled steel tubes which, in turn, are fastened to the aluminum frame by Plexiglas holders. The donor is positioned in the tank so that the detonator axis is normal to the plane in which the test samples are usually placed. This orientation reduces irregularities in the shock front since the arrival of detonation at the donor surface is observed to be more symmetrical in planes normal to the detonator axis. The Teflon capsules are oriented so as to present the flat ends toward the center of the spherical donor to ensure as close to one-dimensional loading of the sample as possible. The explosive test samples are set at different distances from the donor to obtain various input pressures. The sketch in Figure 2 shows the general arrangement of the donor and four capsule holders within the tank of water. More test specimens can be added to the arrangement as indicated by the photograph in Figure 3. The extra capsule holders are mounted in holes drilled at an angle in the PMMA holder which was placed in front of the donor sphere.

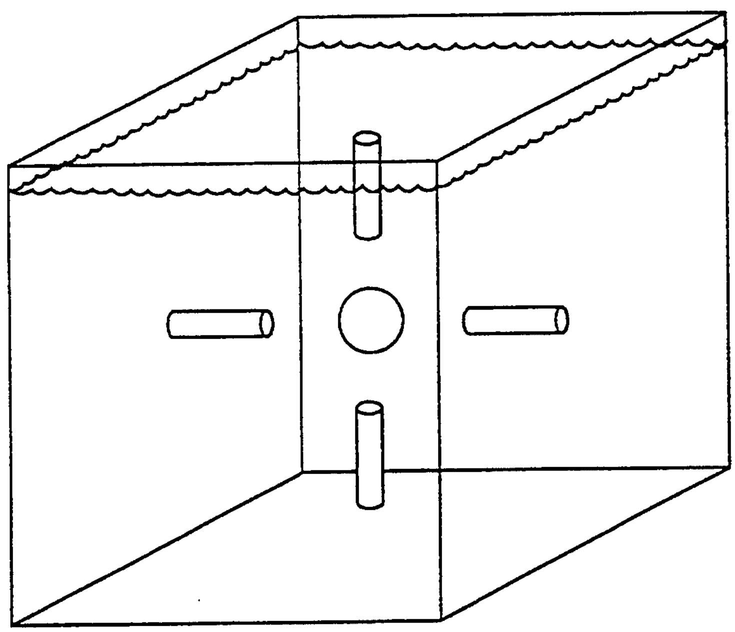


FIGURE 2. CAPSULE PLACEMENT AROUND PENTOLITE SPHERE

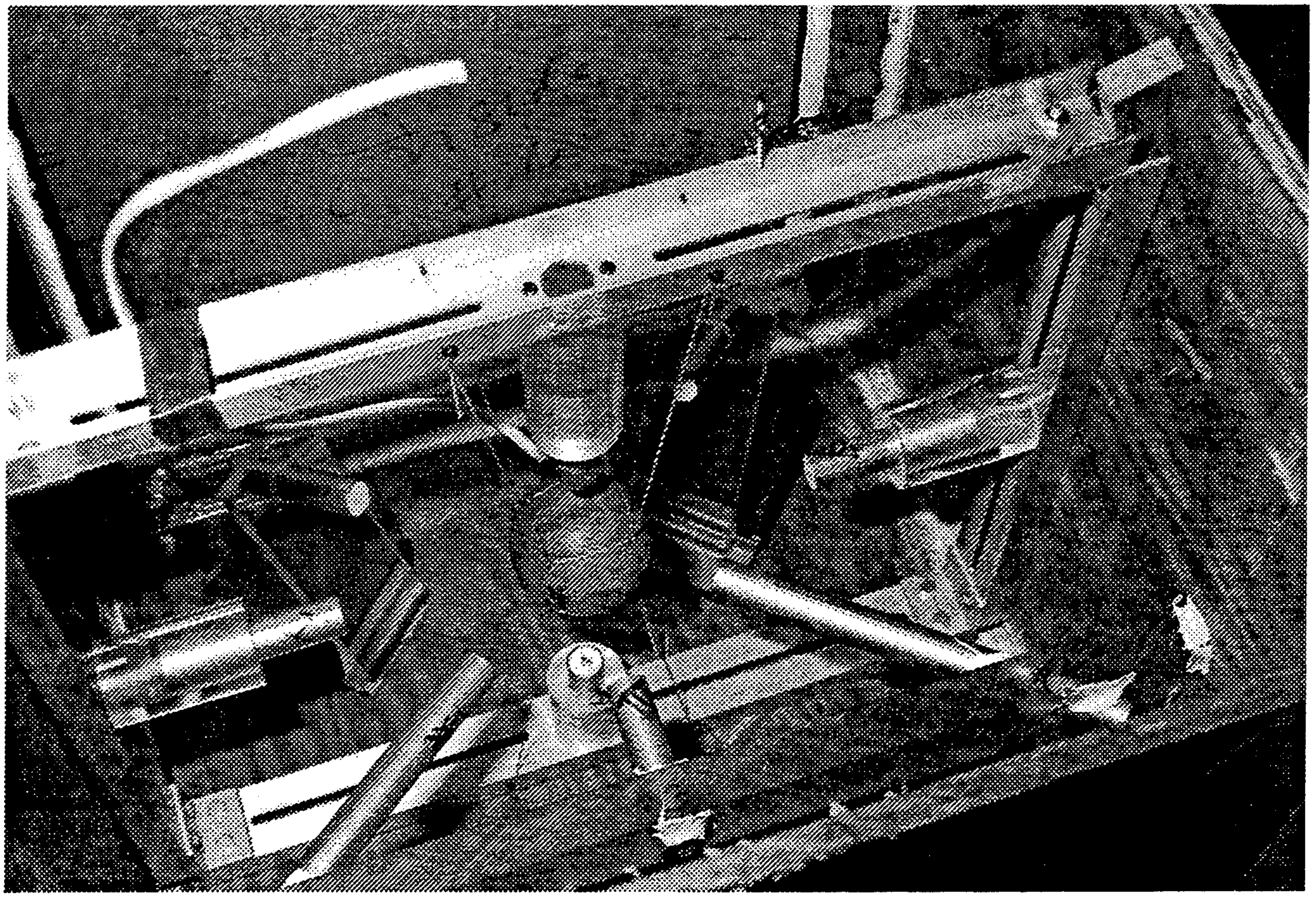
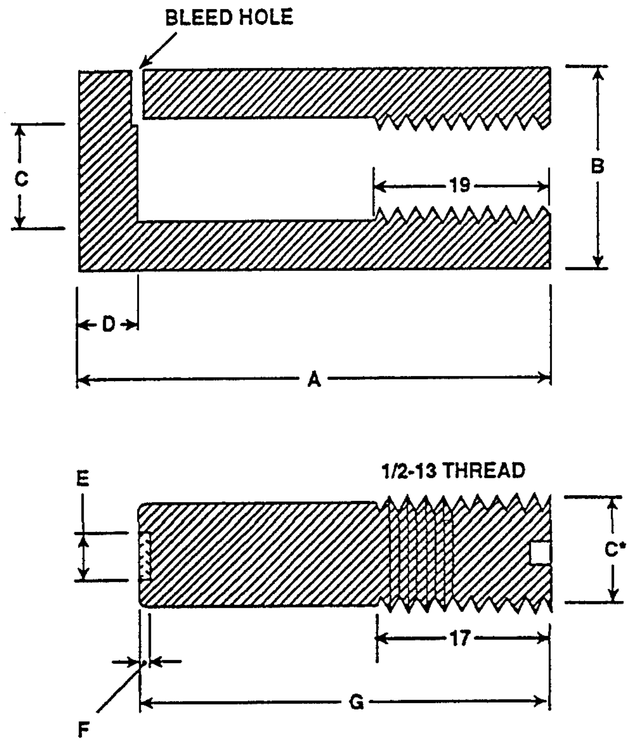


FIGURE 3. PLACEMENT OF NINE TEST SAMPLES IN UNFILLED TANK

**Sample Holder:**

The Teflon sample holder (capsule) is in two parts—a cap and a threaded rod (plug) with a cavity in one end to accommodate the explosive test sample (Figure 4). A 1.6-mm-diameter bleed hole is located at the end of the cavity in the cap to permit air to escape when the threaded plug is inserted into the cap. In early versions of the test capsule, the plug was threaded along its entire length. However, the plug had a tendency to warp, causing it to bind while being screwed into the cap. This made it difficult to know if the explosive specimen was properly confined with the capsule. As a consequence, the threaded length inside the cap was changed to 19 mm (17 mm for the plug). To seal the sample after assembly, the two parts of the capsule originally were etched chemically to permit bonding with special epoxy. Unfortunately, the bonding was so strong that it made it extremely difficult to recover the test sample. The final design uses the 19-mm-long thread without cement. A pipe thread compound containing Teflon is used to seal the threads. After assembly, the bleed hole is plugged with Duxseal.




DIMENSIONS IN MILLIMETERS

| | | | |
|---|---|---|---|
| A | 50.8 | E | 5.2 (dia) |
| B | 21.3 (dia) | F | 0.89 |
| C | 10.71 (dia) | G | 44.5 |
| D | 6.4 | | |

*SNUG FIT

FIGURE 4. TEFLON SAMPLE CAPSULE:
CAP (TOP) AND THREADED PLUG

**Support Tube:**

The steel support tube is bored out to a diameter which just allows the sample capsule to slide within the tube. The reamed section is just deep enough to allow the capsule to be pushed in flush with the edge of the steel tube. The capsule is held in place by the close fit. The steel support tube has several 2.4-mm holes drilled in the side along its length to permit water to fill the tube completely. A PMMA collar can be made so that the steel support tube can be positioned at any desired angle. The steel support tube arrangement is shown in Figure 5. The length of the PMMA collar, as well as the length of the steel tube, can be such that considerable flexibility in adjustment is possible.

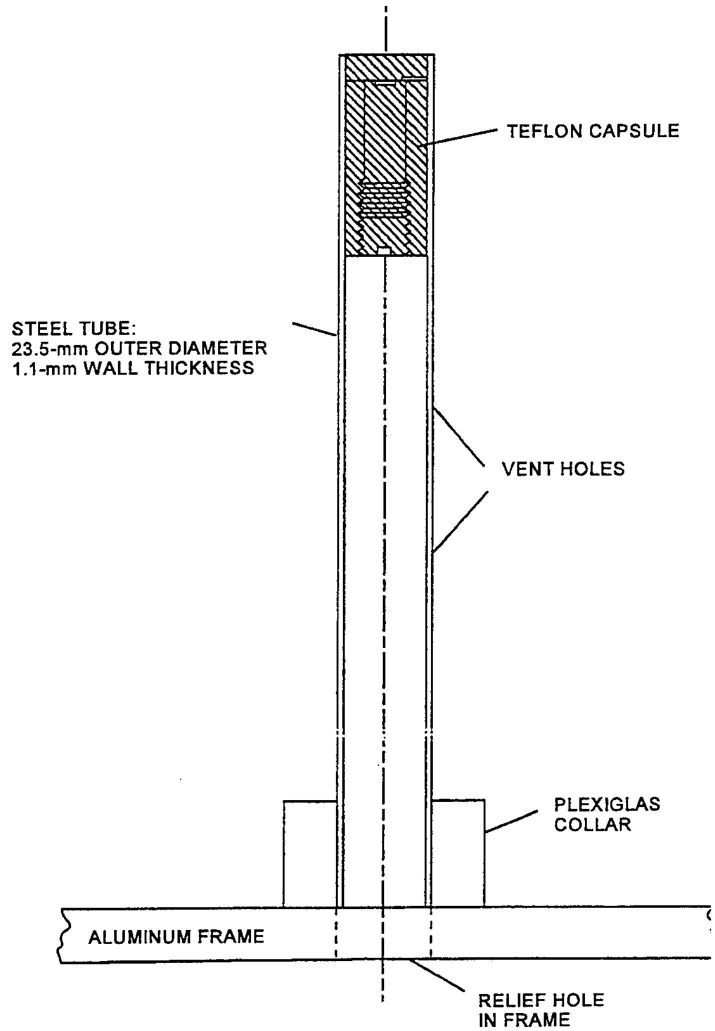



FIGURE 5. CAPSULE HOLDER ARRANGEMENT

**Reduction of Capsule Damage:**

Although a number of improvements in the recovery system were made during the development period, damage to the capsules remained excessive. Toward the end of the investigation, it was suspected that the impact of the tube against the aluminum frame might be the main reason for the excessive damage. Because of this, holes were cut in the aluminum frame to allow the steel tube to slip through the Plexiglas collar when the shock impacted the tube. Relief holes in the aluminum frame can be seen in Figure 3. This apparently solved the problem since all explosive samples were successfully recovered after this alteration was made.

**Shock History of Recovered Sample:**

The UST donor used in the recovery system subjects the explosive samples to relatively long low-pressure shocks of spherical geometry. The shock duration is 20 to 40 μs in the water, the duration increasing with distance from the donor.[3] The calibration of the UST[1], i.e., the peak pressure in the water, $P_w$, as a function of distance from the donor surface, $x$, is given in Table I. The distance is measured along an imaginary straight line extending outward from the center of the spherical donor.

TABLE I. CALIBRATION OF THE UNDERWATER SENSITIVITY TEST

[The water gap, $x_w$, is the sum of a number in the first column and a number in the first row. Pressures in kilobars.]

| $x_w$ (mm) | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 10 | 69.48 | 65.53 | 61.89 | 58.53 | 55.41 | 52.53 | 49.86 | 47.38 | 45.09 | 42.95 |
| 20 | 40.96 | 39.11 | 37.38 | 35.76 | 34.25 | 32.83 | 31.51 | 30.26 | 29.09 | 27.99 |
| 30 | 26.95 | 25.98 | 25.05 | 24.18 | 23.36 | 22.58 | 21.84 | 21.14 | 20.47 | 19.84 |
| 40 | 19.24 | 18.67 | 18.12 | 17.60 | 17.11 | 16.63 | 16.18 | 15.75 | 15.33 | 14.94 |
| 50 | 14.56 | 14.20 | 13.85 | 13.51 | 13.19 | 12.88 | 12.59 | 12.30 | 12.03 | 11.76 |
| 60 | 11.51 | 11.26 | 11.02 | 10.79 | 10.57 | 10.36 | 10.15 | 9.95 | 9.76 | 9.58 |
| 70 | 9.40 | 9.22 | 9.02 | 8.89 | 8.73 | 8.57 | 8.42 | 8.28 | 8.14 | 8.00 |
| 80 | 7.87 | 7.74 | 7.61 | 7.49 | 7.37 | 7.26 | 7.15 | 7.04 | 6.93 | 6.83 |
| 90 | 6.73 | 6.63 | 6.53 | 6.44 | 6.35 | 6.26 | 6.17 | 6.09 | 6.00 | 5.92 |
| 100 | 5.84 | 5.77 | 5.69 | 5.62 | 5.55 | 5.48 | 5.41 | 5.34 | 5.27 | 5.21 |
| 110 | 5.15 | 5.08 | 5.02 | 4.96 | 4.91 | 4.85 | 4.79 | 4.74 | 4.69 | 4.63 |
| 120 | 4.58 | 4.53 | 4.48 | 4.43 | 4.39 | 4.34 | 4.29 | 4.25 | 4.21 | 4.16 |
| 130 | 4.12 | 4.08 | 4.04 | 4.00 | 3.96 | 3.92 | 3.88 | 3.84 | 3.81 | 3.77 |
| 140 | 3.73 | 3.70 | 3.67 | 3.63 | 3.60 | 3.57 | 3.53 | 3.50 | 3.47 | 3.44 |
| 150 | 3.41 | 3.38 | 3.35 | 3.32 | 3.29 | 3.27 | 3.24 | 3.21 | 3.18 | 3.16 |
| 160 | 3.13 | 3.11 | 3.08 | 3.06 | 3.03 | 3.01 | 2.98 | 2.96 | 2.94 | 2.91 |
| 170 | 2.89 | 2.87 | 2.85 | 2.83 | 2.81 | 2.78 | 2.76 | 2.74 | 2.72 | 2.70 |
| 180 | 2.68 | 2.66 | 2.65 | 2.63 | 2.61 | 2.59 | 2.57 | 2.55 | 2.54 | 2.52 |
| 190 | 2.50 | 2.48 | 2.47 | 2.45 | 2.43 | 2.42 | 2.40 | 2.39 | 2.36 | 2.35 |
| 200 | 2.34 | — | — | — | — | — | — | — | — | — |

Highly instrumented experiments would be required to determine the shock pressure-time profile of the small explosive test samples to an uncertainty of ±5%. A second approach to obtaining the stress-time profiles to uncertainties of ±10% would be to run two-dimensional hydrocodes. Since neither of the above approaches have been used, all that can reasonably be done is to determine the peak pressure entering the test samples to uncertainties of ±10% and shock pulse time half-widths to ±20% (see Appendix G of ref. 1).

An example of how the approximate shock history in an explosive sample is determined is as follows. First, the peak pressure in the water, taken from Table I, is plotted as a function of $x$, as in Figure 6. It is seen that the peak pressure of the spherical shockwave in water is 14.2 kbar just before the shock front contacts the center of the flat surface of the Teflon capsule at $x_1$ = 50.8 mm. This stress is found to be 19.9 kbar by using standard impedance relations and is shown in Figure 7 for a graphical solution. The shock impedance relationships (shock velocity versus particle velocity) for water and Teflon are taken from ref. 5 and 6 respectively. The peak stress entering the Teflon at $x_1$ is known to an uncertainty of ±8% according to standard error analysis[4]. Next, the stress in the Teflon capsule at the boundary with a TATB sample prior to the wave entering the TATB, $x_2$ = 57.2 mm, is 17.0 kbar to within

±10%. It is assumed that the decay of the peak pressure through 6.4 mm of Teflon is proportional to the decay of pressure through an equivalent distance in water, as demonstrated in Figure 6. Finally, the peak pressure entering the TATB sample, 19.0 kbar ± 15%, is determined by using the unreacted shock impedance relation for TATB.[7]

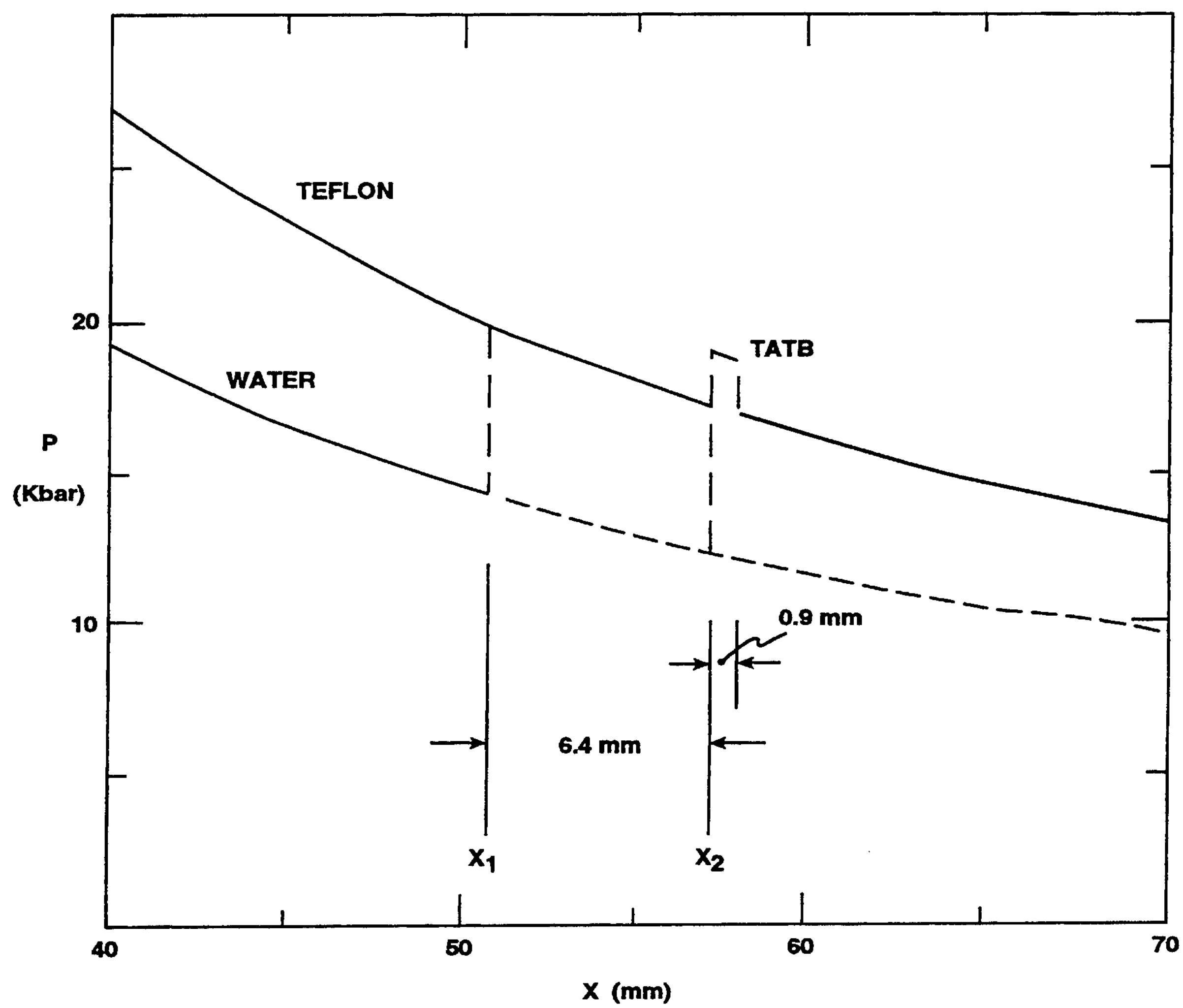



FIGURE 6. METHOD OF DETERMINING THE PEAK STRESS RESEARCHING TO TEFLON AND TATB

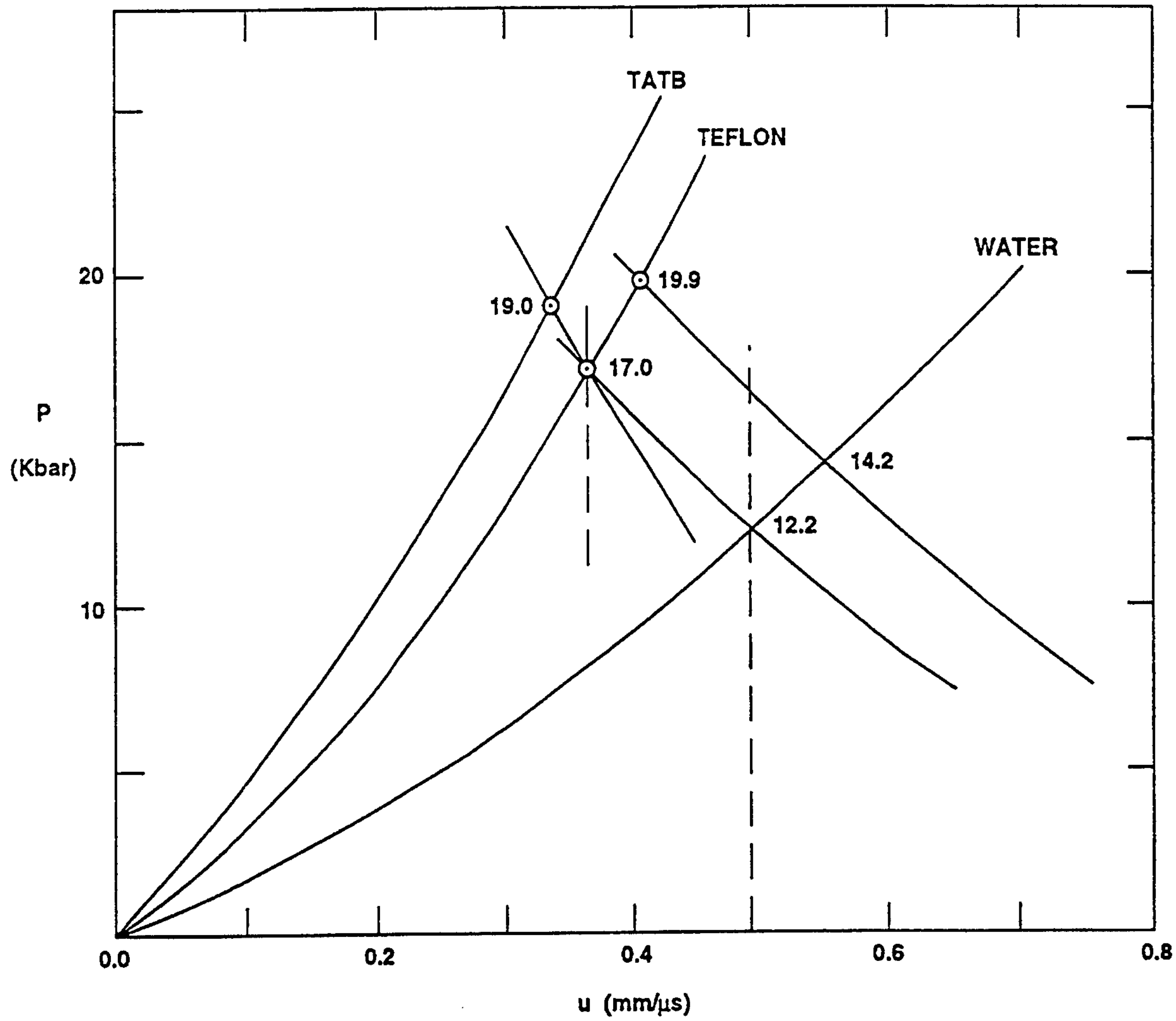



FIGURE 7. GRAPHIC METHOD OF DETERMINING PRESSURES TRANSMITTED TO THE TEFLON CAPSULE AND TATB SAMPLE

Since the Teflon and the explosive have about the same shock impedance, the shape of the stress-time pulse at the center line of the small sample (0.9 mm thick by 5 mm diameter) is primarily limited by the relief waves originating from the Teflon capsules. Assuming the pressure pulse half-width is about equal to the time of travel of the relief wave from the outer radial edge to the center of the Teflon holder gives a pulse half-width of about 5.3 µs (i.e.~ 10.6 mm/[2 mm/µs]).

## RESULTS

In Table II are the results of the recovery experiments done to date. A number of papers reported the chemical and physical changes found in some of the samples in the table that were shocked to threshold pressures.[8,9,10,11] In recovered shocked TATB, sub-micron ragged holes were found, accompanied by a fine deposit of furoxan and furazan derivative of TATB. Since the furoxans are more sensitive than TATB, this alteration of the molecule provides a chemical basis of *hot spot* formation and sensitization of the explosive. The furazans are produced by the formation of a water molecule and identified as the first of the exothermic steps in the decomposition reaction of the molecule. In RDX and HMX, the products generated by shock compression are mostly volatile. Analysis of the recovered nitramine material observed a loss of nitro functional groups. In HMX subjected to an underwater shock, a 16% loss of nitro groups was observed.

TABLE II. RESULTS OF RECOVERY EXPERIMENTS

| Experiment No.[a] | Sample | Distance capsule is from sphere (mm) | Pressure in Teflon (Kbar) | Results |
|---|---|---|---|---|
| 83-77A | TATB | 135 | 4.9 | Sample reacted, no recovery |
| 83-79 | TATB | 125 | 5.5 | Sample recovered |
| 83-81 | TATB(A) | 76 | 10.3 | Sample recovered |
| | TATB(B) | 64 | 13.1 | Sample recovered |
| | TATB(C) | 57 | 14.9 | Sample recovered |
| | TATB(D) | 51 | 17.3 | Sample recovered |
| 88-665 | HMX(A) | 159 | 4.2 | Sample recovered, three pieces found |
| | HMX(B) | 182 | 3.5 | Sample recovered, three pieces found |
| | HMX(C) | 129 | 5.1 | Sample recovered, three pieces found |
| | HMX(D) | 130 | 5.1 | Two pieces found |
| | NTO(E) | 67 | 12.4 | Sample recovered intact |
| | NTO(F) | 49 | 18.1 | Sample recovered intact |
| 92-R1 | TNT(I) | 100 | 7.4 | Sample recovered |
| | TNT(II) | 120 | 5.7 | Sample recovered |
| | TATB(III) | 50 | 17.7 | Sample recovered |
| | TATB(IV) | 40 | 22.8 | Sample recovered |
| | RDX crystals(VI) | 50 | 17.7 | End blown off capsule, small parts of crystals found |
| | RDX crystals(VII) | 40 | 22.8 | End blown off capsule, no recovery |
| | CL20/Oil(VIII) | 100 | 7.4 | Capsule recovered, soot around air hole, residue found |
| | CL20/Oil(IX) | 150 | 4.5 | Capsule recovered, soot around air hole, residue found |

[a]TATB, TNT, NTO, and HMX samples were pressed to 95% to 97% theoretical maximum density.

## SUMMARY

A technique has been developed to recover explosives undergoing stresses as high as 26 kbar. Physical (microscopic) and chemical examination of recovered explosives has led to the discovery of new chemical reactions occurring in these explosives.

Improved understanding of this recovery technique results requires two-dimensional hydrocode calculations. These calculations will also give guidance for future improvements of this recovery technique. One obvious improvement would be to replace the steel capsule holders with plastic ones to reduce the impedance mismatch between the capsule holders and the Teflon capsules.

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PO BOX 3707
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RIDGECREST CA 1

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CHAMBERLAIN MFG CORP
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HERCULES
BACCHUS WORKS
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MAGNA UT 84044 1

HERCULES
BACCHUS WORKS
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MAGNA UT 84044 1

HERCULES
BACCHUS WORKS
ATTN L LOSEE
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BACCHUS WORKS
ATTN T SPEED
MAGNA UT 84044 1

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HARTWELL ROAD
BEDFORD MA 01730 1

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A DIVISION OF MAXWELL LABS INC
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PO BOX 1620
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BROOKLYN PARK MN 55428 1

ALLIANT TECHSYSTEMS INC
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ATTN A CUMMINGS ET BLDG X59
KENT TN14 7BP
UNITED KINGDOM 1

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KENT TN14 7BP
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ALDERMASTON
READING BERKSHIRE RG7 4PR
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READING BERKSHIRE RG7 4PR
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ALDERMASTON
READING BERKSHIRE RG7 4PR
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ARDEER SITE
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MATERIAL RESEARCH LABORATORY
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ATTN R STENSON (S DIV)
EMPRESS STATE BUILDING
LILLIE ROAD LONDON SW6 1TR
UNITED KINGDOM 1

ICI EXPLOSIVES
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STEVENSTON KA20 3LN
UNITED KINGDOM 1

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ORLANDO TECHNOLOGY INC
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DE VAUJOURS-MORONVILLIERS
ATTN J N OECONOMOS
B P N° 7 77181 COURTRY
FRANCE 1

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CENTRE D'ETUDES
DE VAUJOURS-MORONVILLIERS
ATTN F CHAISSE
B P N° 7 77181 COURTRY
FRANCE 1

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| DEFENSE RESEARCH ESTABLISHMENT<br>VALCARTIER<br>ATTN ROCCO FARINACCIO<br>PO BOX 8800<br>COURCELETTE QUEBEC<br>CANADA<br>GOA 1R0 | 1 |
| DEFENSE RESEARCH ESTABLISHMENT<br>VALCARTIER<br>ATTN IRENE HOOTON<br>PO BOX 8800<br>COURCELETTE QUEBEC<br>CANADA<br>GOA 1R0 | 1 |
| DEFENSE RESEARCH ESTABLISHMENT<br>VALCARTIER<br>ATTN GRANT MC INTOSH<br>PO BOX 8800<br>COURCELETTE QUEBEC<br>CANADA<br>GOA 1R0 | 1 |

**Internal:**

| | |
|---|---|
| TDE (S MITCHELL) | 3 |
| 102 | 1 |
| 40D (E JOHNSON) | 1 |
| 40 | 1 |
| 410 (R GRANDE) | 1 |
| 410 (R GARRETT) | 1 |
| 410 (W FURR) | 1 |
| 410 | 1 |
| 450 (O PARRENT) | 1 |
| 450 (L LIPTON) | 1 |
| 460 (J GASPIN) | 1 |
| 460 (G HARRIS) | 1 |
| 460 R TUSSING) | 1 |
| 460 (R BARASH) | 1 |
| 460 (T FARLEY) | 1 |
| 460 (W MCDONALD) | 1 |
| 460 (H MAIR) | 1 |
| 90I | 1 |
| 90H | 1 |
| 90 | 1 |
| 90E | 1 |
| 90D | 1 |
| 90C | 1 |
| 910 (Y TRAN) | 1 |
| 910 (SITZMANN) | 1 |
| 910 | 1 |
| 912 (D CICHRA) | 1 |
| 920 | 1 |
| 930 | 1 |
| 940 | 1 |
| 941 (A DUONG) | 1 |
| 941 (C GOTZMER) | 1 |
| 941 (L NOCK) | 1 |
| 941 (E ANDERSON) | 1 |
| 950 (L MONTESI) | 1 |
| 950 (M SWISDAK) | 1 |
| 952 (J LAIB) | 1 |
| 954 (L DAVIE) | 1 |
| 8510 | 3 |
| 8530 | 3 |
| 920C1(R BERNECKER) | 1 |
| 920C2 (J FORBES) | 9 |
| 9210 (P MILLER) | 1 |
| 9210 (H JONES) | 1 |
| 9210 (C COFFEY) | 1 |
| 9210 (R GUIRGUIS) | 1 |
| 9210 (F ZERILLI) | 1 |
| 9210 (D WOODY) | 1 |
| 9220 (W WILSON) | 1 |
| 9220 (C RICHMOND) | 1 |
| 9220 (D TASKER) | 1 |
| 9220 (T LIDDIARD) | 1 |
| 9220 (P GUSTAVSON) | 1 |
| 9220 (RICHARD LEE) | 1 |
| 9220 (R BAKER) | 1 |
| 9230 (G SUTHERLAND) | 1 |

IHTR 1821

| | |
|---|---|
| 420 (B PARK) | 1 |
| 420 (W WALKER) | 1 |
| 420 (A DARE) | 1 |
| 420 (H CHEN) | 1 |
| 420 (D BETANCOURT) | 1 |
| 420 | 1 |
| 9230 (E LEMAR) | 1 |
| 9230 (J DAVIS) | 1 |
| 9240 (B GLANCY) | 1 |
| 9311 (K NEWMAN) | 1 |
| 9410K (J CHANG) | 1 |
| 9410 (P DENDOR) | 1 |

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